

CLAIMS

We claim:

1. An analytical system for measuring total nitrogen and/or total sulfur in a sample, comprising:

a furnace system for oxidizing the sample, the furnace system having an inlet and an outlet and producing a combustion gas stream containing nitric oxide (NO) and/or sulfur dioxide (SO₂), the furnace system being capable of operating at about 900°C or higher;

a sample introduction apparatus for delivering the sample to the inlet of the furnace system;

an electronic mass flow controller to regulate flow from a source of high-purity oxygen, the oxygen being provided for oxidizing the sample;

an electronic mass flow controller to regulate flow from a source of carrier gas into the furnace system;

a dehydration unit in fluid communication with the outlet of the furnace system for removing water from the combustion gas stream;

a catalytic combustion gas converter system in fluid communication with the dehydration unit, comprised of an inert tube containing a graphite catalyst capable of converting NO₂ and/or NO_x to NO without changing the ambient concentration of the SO₂ in the combustion gas stream, a heating mantle, a power source, and gas tubing fittings, the catalyst being heatable to between 300° C to 400° C when receiving a gas flows of 300 to 500 mL/min from said dehydration unit,

a first electrochemical detector having an electrolyte, a sensing electrode, a counter electrode and a reference electrode, the electrochemical detector being sealed in a container and having a membrane for receiving a portion of the combustion gas stream from the dehydration unit, wherein electrochemical detector is adapted for selectively detecting NO or SO₂ in the combustion gas stream.

2. The analytical system of claim 1, further comprising a computer control system coupled to the furnace system, the sample introduction apparatus, the mass flow controllers for oxygen and carrier gas, and the detector for providing supervisory control, monitoring, data storage and analysis, computer control system being capable of providing

control for the furnace system, the sample introduction apparatus, the mass flow controllers and the detector.

3. The analytical system of claim 1, further comprising a second electrochemical detector operated in series with the first electrochemical detector, wherein a chemical compound measured by the second electrochemical detector does not interfere with the first electrochemical detector.

4. The analytical system of claim 2, wherein the system is adapted for measuring total nitrogen and total sulfur in a liquid hydrocarbon sample.

5. The analytical system of claim 1, wherein the catalyst is graphite.

6. The analytical system of claim 1, wherein the catalyst is graphite powder.

7. The analytical system of claim 1, wherein the detector detects nitric oxide (NO), further comprising a detector for detecting sulfur dioxide (SO₂), wherein the detectors are capable of operation in series.

9. The analytical system of claim 1, wherein the detector detects nitric oxide (NO), and wherein the analytical system is capable of determining total nitrogen according to ASTM designation D6366-99.

10. The analytical system of claim 9, further comprising a detector for detecting sulfur dioxide (SO₂), wherein the analytical system is capable of determining total sulfur according to ASTM designation D6428-99.

11. The analytical system of claim 10, wherein the detectors are capable of operation in series.

12. An analytical system for measuring total nitrogen and total sulfur in a liquid hydrocarbon sample, comprising:

a furnace system capable of operating at about 900°C or higher to receive and oxidize the sample, the furnace system having an inlet and an outlet and capable of

producing a combustion gas stream containing nitric oxide (NO) and/or sulfur dioxide (SO₂);

a sample introduction apparatus for delivering the sample to the furnace system;

a first mass flow controller for regulating flow of a carrier gas to the sample introduction apparatus;

a second mass flow controller for regulating flow of oxygen to the inlet of the furnace system;

a dehydration unit in fluid communication with the outlet of the furnace system for removing water from the combustion gas stream;

a catalytic combustion gas converter system in fluid communication with the outlet of the dehydration unit, comprised of an inert tube containing a bed of graphite catalyst, a heating mantle, a power source, and gas tubing fittings, the bed of graphite catalyst being heatable to between 300° C to 400° C when receiving a gas flows of 300 to 500 mL/min from said dehydration unit,

at least two electrochemical detectors adapted to operate in series, each electrochemical detector having an electrolyte, a sensing electrode, a counter electrode and a reference electrode, the electrochemical detector being sealed in a container and having a membrane for receiving a portion of the combustion gas stream from the dehydration unit, said at least two electrochemical detectors selectively detecting NO and SO₂ in the combustion gas stream; and

a control system coupled to the furnace system, the sample introduction apparatus and each electrochemical detector for providing supervisory control, monitoring, data storage and analysis.

13. A method capable of determining total nitrogen and/or total sulfur in a liquid sample, the method comprising the steps of:

providing a pyrolysis furnace capable of vaporizing the sample;

providing a sample introduction system for introducing the sample to the furnace;

providing a quartz combustion tube capable of operating at 900°C, the combustion tube being adapted for combusting the sample;

producing a combustion gas as an exhaust from the combustion tube;

drying the combustion gas to form a dry sample gas;

passing the dry gas sample over a graphite catalyst heated to a temperature of 300° C to 400° C,

detecting a compound in the dry sample gas which has passed over said graphite catalyst using an electrochemical detector having at least three electrodes; and

determining total nitrogen and total sulfur based on measurement of the detected compound.

15. The method of claim 14, wherein at least two detectors are operated in series.

16. The method of claim 13, wherein total nitrogen is determined in conformance with a standard set forth by the American Society for Testing and Materials (ASTM) in designation D6366-99.

17. The method of claim 16, wherein total sulfur is determined in conformance with a standard set forth in ASTM designation D6428-99.